TITLE

HOLDER FOR SAMPLE MATERIALS USED IN HIGH THROUGHPUT PHYSICAL VAPOR DEPOSITION MATERIAL STUDIES

RELATED APPLICATION

[0001] This is a continuation-in part of our co-pending application Serial No. 10/757,302 filed on January 14, 2004 entitled "HIGH THROUGHPUT PHYSICAL VAPOR DEPOSITION SYSTEM FOR MATERIAL COMBINATORIAL STUDIES."

FIELD OF THE INVENTION

[0002] The present invention relates to a holder device, particularly a substrate sample holder useful with a high throughput systems for the synthesis of materials, such as heterogeneous catalysts and other materials synthesized by the combinatorial method in processes such as physical vapor deposition (PVD). After synthesis, the samples are tested for utility as a catalyst or other application.

BACKGROUND AND SUMMARY OF THE INVENTION

[0003] Our co-pending application, is incorporated herein by reference and discusses difficulties involved in the discovery of material compositions, such as catalysts, from among almost limitless material or compositional possibilities, layer configurations, and material proportions. When samples are formulated and produced, one by one, for testing, using expensive and complex equipment and repetitive test protocols considerable time is consumed. The co-pending application discloses a high throughput system for synthesizing a group of material compositions

in a same batch using multiple programmable plasma gun clusters and a positionally programmable x-y table enclosed in a PVD chamber.

[0004] It is an object of this invention to provide a sample holder for use in a programmable matrix high throughput system to allow the essentially simultaneous fabrication of multiple, different combinatorial catalyst samples in a batch, in a system in which predetermined spots, substrates or electrodes on a relatively planar array configuration are separately coated with multiple material constituents.

[0005] The invention is described more fully in the following description of the preferred embodiment considered in view of the drawings in which:

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

[0006] Figure 1 is a side cross section view of a multiple plasma gun cluster focused toward a substrate assembly having an array of separate spot areas intended to be coated with different material combinations in a PVD chamber. In a programmable system, each sample spot in the substrate array is oriented with respect to a plasma beam and a mask and a moveable x-y table.

[0007] Figure 2 is a plan view of multiple coating spots maintained as a group array in a substrate block assembly of the invention shown in Figures 3, 4, 5, and 6.

[0008] Figure 3 is a side cross section view of a multiple plasma gun cluster focused toward a substrate holder block of the invention in which the array of separate spot areas intended to be coated with different material combinations in a PVD chamber comprises an assembly of discrete rods maintained in the block.

[0009] Figure 4A and Figure 4B are respectively a perspective front side view and a front side assembly view of an example of a substrate block holder for maintaining a plurality of substrate samples in the system.

[0010] Figure 5A and Figure 5B are respectively a perspective back side view and a back side assembly view of an example of a substrate block holder for maintaining a plurality of substrate samples in the system.

[0011] Figure 6 is a cross section view of the assembly configuration in the block for maintaining each separate rod in a predetermined spot area fixed in position in the block during the synthesis process.

DETAILED DESCRIPTION OF THE INVENTION

[0012] The invention described in our co-pending application is an infinitely variable physical vapor deposition matrix system that allows the fabrication of multiple combinatorial material samples by the co-deposition of multiple materials or the sequential layer by layer deposition of multiple constituents. Using the system of the invention, the optimum mix or combination of materials for a pre-determined application can be determined by subsequent testing and evaluation of the samples produced.

[0012] In the high throughput system using a PVD process, a substrate or substrate holder having an array of discrete spots or coating areas is introduced into a PVD chamber. As shown in Figure 1, the chamber includes more than one multiple group, cluster, or arrays of plasma guns 10 directed toward a substrate assembly 14, preceded by mask 11 having opening 12. For clarity, in the side view of Figure 1, only one gun cluster 10 is shown. Substrate assembly 14 is moveable in an x-y

plane within the chamber with respect to the plasma gun array 10. As the substrate assembly 14 is aligned facing plasma gun array 10, the plasma beam 20 passes through mask opening 12 and deposits a pre-programmed mixture or proportion of materials, (co-deposition or sequential layers) upon pre-determined discrete areas, 14a, 14b, 14... in an array of discrete spots on a relatively planar surface. The relative x-y movement of the substrate 14 is controlled by appropriately programming orthogonal locations, X1, Y1, X2, and Y2 for each spot on the substrate with respect to the programmable plasma beam in a x-y table 15. [Rotational movement in an axial Z direction is not an attribute of the present invention.]

[0013] In an example of operation of the system, the relative radial position of the substrate with respect to plasma gun positions is controlled by an appropriate programming and control means. Once the substrate is aligned in position with respect to a plasma gun cluster, a predetermined area or electrode or spot area in an array, such as 14a, 14b or 14..., on the substrate or holder is aligned with respect to the opening in the mask 12 by a programmed positioning of the substrate with respect to an x-y axis by an x-y table. The rate or quantity of material deposition with respect to a specific area on the substrate in determined by controlling the shutter, power and other operating parameters of the individual guns.

[0014] The present invention comprises a holder for maintaining substrates in an array in a system in which the substrates are subjected to a high throughput materials deposition. The holder is a block assembly comprising a face plate, middle plate and retainer plate attached in sequence and aligned in an axis in which a plurality of cylindrical substrates are maintained in cylindrical chambers formed in an

array with respect to the face surface of the block assembly. The substrates are positioned within the chambers by a spring mechanism during the processing of the substrates, and removable therefrom after the completion of processing. The holder is secured to a programmable x-y table in a PVD process in a relationship in which the focus of the PVD plasma source and the surfaces of the substrates maintained in the holder are in approximately the same plane. Examples of the array include substrates arranged in columns and rows in a matrix in the block assembly; the relationship of the number of substrates in rows to the number of substrates ($_N$) in columns may be $rows_N = columns_N$; the relationship of the number of substrates in one column to of the number of substrates in an adjacent column may be substrates in $column_N = N$ and substrates in column $N+1 = N+1$; the relationship of the number of substrates in one row to of the number of substrates in an adjacent row may be substrates in $row_N = N$ and areas in row $N-1 = N-1$. Generally, however, the substrates may be arranged in any array such that the location of each substrate in the array is capable of being positioned with respect to a defined point by the manipulation of a programmable x-y table.

[0015] Substrates maintained in the block in a PVD process are longitudinally extending cylindrical electrodes maintained in cylindrical columns in the block. The upper surface of the electrode is inset within the block such that the transverse cross-section area of an opening in the upper surface of the column in the block in which the electrode is positioned is less than the transverse cross-section area of the upper surface of the electrode. A secondary mask may be positioned within a column in the block adjacent the upper surface of the electrode in the column; and a retainer and

spring may be positioned within a column in the block between the lower surface of the electrode in the column and the back retainer plate.

[0016] Substrate holder assembly of the invention 46 is shown in Figure 3 in a detail side view. The holder maintains individual sample element electrodes, rods or substrates in an array in a block or holder, such as 46a, 46b and 46..., on which candidate materials are deposited. The holder is affixed to the support plate of an x-y table 44. Mask 41 having opening 42 is interposed between the holding block 46 and plasma gun array 45 which generates the plasma beam 50. The relative positions of the substrate sample elements 46a, 46b and 46... maintained in the block with respect to the mask 41 and plasma beam 50 are moveable with respect to each other during the deposition process. Typically, the plasma guns are fixed at radial positions at the perimeter of a PVD chamber. Holding block 46 and support plate 44 may be fixed or moveable with respect to one another, dependent upon the relationship of each to an x-y table. The mask opening may be in a shape and size determined by the system operator; and the mask may include more than one opening.

[0017] Figure 2 illustrates, in a plan view from the top, a substrate 14 having a four (4) element by four (4) element array of deposition or coating spots intended to be exposed to programmed plasma beams and their respective material constituents. In the array, the sixteen (16) individual spot areas are identified as 14a, 14b, 14...; each spot position may correspond to an electrode mounted in a holding block. The spot positions of the substrate identified in the array are each individually exposed to plasma from one or more guns in the chamber, by controlling the position of the

holder, and consequently, a specific area on the substrate, with respect to the fixed mask and plasma beam[s] by appropriately programming the locations on the x-y table on which the holder/ substrate is mounted. In Figure 2, the substrate is shown after a high throughput deposition coating treatment; each of the sixteen (16) individual spots or electrodes, namely 14a, 14b, 14..., has deposited thereon a different co-deposition or layer sequence as determined by programming and control means.

[0018] Figure 4A and Figure 4B illustrate a substrate holder assembly 46, also shown in Figure 3 which illustrates the holder function during the coating process. Assembly 46 in Figure 4A includes, front face plate 71, block or middle plate 46M, and back retainer plate 73. In the assembly view of Figure 4B, screws or fasteners 72 maintain the front face plate secured to the middle plate 46M. Openings in a matrix corresponding to the arrangement of samples 46a ... 46... in the block are included in the face plate essentially concentric with the sample arrangement; typically the openings are sized in diameter less than that the diameter of the samples, securing the samples in the block by the spring loading mechanism described below. The openings may include an inset from the upper face of the sample and a surface chamfer, thus acting as a second mask directing the plasma beam to the surface of the rod or electrode during the deposition process. Back retainer plate 73 of the bock is secured to the holder block by screws or fasteners 74.

[0019] In achieving high throughput using a programmable x-y table to control position of the substrate holder, and consequently the position of individual substrate samples, the columns and rows of sample areas are preferably arranged in a matrix.

For example, the number of sample areas in rows may equal the number of sample areas in columns: 3 x 3; 4 x 4; 6 x 6; 8 x 8; *etc.* Alternatively, the electrodes in the columns and rows may be staggered, for example, 3-4-3-4; 8-7-8-7-8-7-8, in an order in which the relationship of the number of sample areas in one column to the number of sample areas in an adjacent column is: sample areas in row N = X; sample areas in row N + 1= X+1; or sample areas in column N = X; sample areas in column N + 1= X+1. As long as positions of the sample spot areas on the substrate surface are programmable, the specific matrix array arrangement of the holder block is discretionary with the test protocol designer. Utilizing a control system interrelating the operation of the plasma guns in the clusters with respect to the substrate areas, separately controllable plasma sources can be programmed such that the plasma materials are deposited either as a layer-by-layer deposition or in a co-deposition relationship, or both.

[0020] Figures 4A and 4B and 5A and 5B respectively show, from the front and back, the assembly of the holder. The holder comprises a face plate 71 attached to block or middle plate 46M to which retainer plate 73 is also attached. The openings in the face plate, 101a, 101b, 101..., essentially corresponding to the spot array to be coated, are concentrically aligned with open cylindrical columns 102a, 102b, 102... in the block or middle plate 46M which receive the electrodes, rods or substrate elements 46... Thus, the block holds each substrate in an array in a pre-arranged pattern. In Figure 5B, an electrode mask 75 is shown positioned in the circular column at the front face of electrode 46.... At the back of the electrode, retainer 76

and spring 77 are shown forcing the sample to the upper surface in the assembly. Pins or dowels 78 assist in maintaining the alignment of the plates 71 and 46M.

[0021] Figure 6 shows a cross section of the holder detailing an individual mechanism in the overall array for securing an individual electrode in the holder assembly showing the configuration of the elements: face plate 71 and fastening screws 72 attaching to block or middle plate 46M and retainer or rear plate 73 with fastening screws 74. The substrate 46... is shown maintained in the column in the middle plate 102... which is concentrically aligned with opening 101... in the face plate. The spring 77 and electrode retainer 76 exert a force upon the electrode to secure the electrode at the spot area 80 at which the surface of the electrode is exposed to the plasma beam during the coating process. Small mask 75 in the opening 101... is at the base of a chamfer or bevel 71C in the opening 101... in faceplate 71. Thus the upper section of the electrode substrate 46... is inset into the face plate 71. A small oversize tolerance in the column 102... in middle plate 46M facilitates the insertion of the electrode. Large mask 90 overlays the face plate opening 101...

[0022] The holder is conventionally sized and formed from metal or ceramic material(s) otherwise capable of withstanding the environment of a PVD deposition process over multiple uses. One electrode, or substrate, is loaded into each chamber in the array of columns in the holder block, in the assembly order shown in Figure 6. Once assembled and secured in the holder block, the block array is appropriately attached to the programmable x-y table and subjected to the high throughput deposition process.

[0023] Having described the invention in detail, those skilled in the art will appreciate that, given the present disclosure, modifications may be made to the invention without departing from the spirit of the inventive concept herein described. Therefore, it is not intended that the scope of the invention be limited to the specific and preferred embodiments illustrations as described. Rather, it is intended that the scope of the invention be determined by the appended claims.